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A JOINT UK-WEST GERMAN MEETING DISCUSSES ADVANCED
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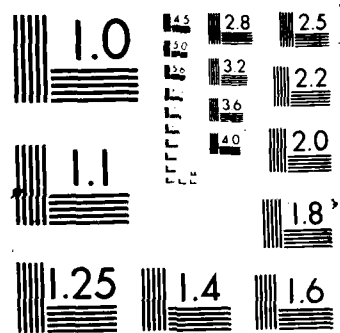
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Louis Cartz

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A JOINT UK-WEST GERMANY MEETING DISCUSSES ADVANCED ENGINEERING CERAMICS

1 INTRODUCTION

A meeting on "High Technology in the Ceramic Industry" was held in April 1987 at Eliot College, the University of Kent, Canterbury. This was a joint meeting of the UK's Institute of Ceramics, and the Deutsche Keramische Gesellschaft EV. The basic science section organized a session on "Advanced Engineering Ceramics" in which about a dozen papers were presented, half from Germany and half from the UK.

The introductory session was devoted to ceramic research in Japan, and this was in fact a general discussion and description of the Japanese approach to industrial procedures and applied research. Two talks were given, both very well presented and, indeed, fascinating. J. Lorrman (GEC Telecommunication Ltd., UK) talked about the Japanese approach to engineering education (Lorrman, 1986). P.A. Evans, University of Leeds, UK, discussed ceramics research in Japan. In both cases, considerable insight was given of the Japanese industrial scene.

Several engineering ceramic systems were discussed at the meeting and these included:

- o The glassy phase in silicon nitride ceramics and sialons,
- o Chemical compatibility of ~~SiC-sialon~~ ^{SiC-sialon},
- o Gas pressure sintering,
- o Oxidation problems of nitrogen ceramics,
- o Transparency of nitrogen ceramics.

2 SILICON NITRIDE CERAMICS AND SIALONS

P. Greil (Max Planck Institut, Stuttgart, West Germany) reviewed the high-temperature behavior of silicon nitride ceramics. There is a considerable drop in strength at $\sim 800^\circ\text{C}$ due to Si-oxy-nitride glass, which is present from the use of sintering additives, and softens at that temperature. The mechanical properties at high temperature of silicon nitride ceramics is very dependent on the

glassy phase present at the grain boundaries (GB) so that this GB glassy phase needs to be controlled. This requires using the minimum of sintering additives, changing the wettability of the glassy phase at the GB, and avoiding impurity segregation at the GB. Further improvement to mechanical properties is by forming composite-type structures. Greil described how many of these factors can be undertaken.

One method of removing the glassy phase from the GB is to reduce the wettability of the glass to the microstructure. When this is done by compositional changes, the glass becomes located at the triple points (TP) between several grains rather than along the GB. For sialon, the amount of glassy phase along the GB and at TP changes with oxygen content in the glass phase; transmission electron microscope (TEM) studies show that the amount of glass at the TP increases with increase of oxygen content.

Another method of obtaining zero GB glass phase is to change the composition of the sialon to the condition where no glassy phase exists. This is the case for the quaternary system Si-Be-O-N and for the quinary systems (Be, Mg, Y, Li)-Si-Al-O-N. The creep rate is much better in Be-sialon where no glass phase exists than for hot-pressed Si_3N_4 (HPSN).

The glassy phase can also be removed by causing the glass to crystallize to form refractory compounds. One way is to add Y_2O_5 , resulting in the formation of metastable $\text{Y}_2\text{SiAlO}_5\text{N}$, which has a composition rather close to the glassy phase composition. In this way the modulus of rupture (MOR) and the other mechanical properties are improved. However, on cooling, more than one phase may form in which differential thermal expansion can cause cracking.

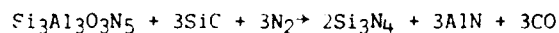
Composites are formed by the addition of whiskers to Si_3N_4 . Geil mentioned that considerable work is in progress in Japan and in France. However, it is necessary to avoid degeneration of the whiskers when forming homogeneous dispersions. Composites under examination by Geil include sintered silicon nitride (SSN) with polycrystalline tetragonal

zirconia with Y_2O_5 (YTZP), SSN with yttrium aluminate garnet (YAG) and HPSN with spinel.

Geil concluded that improved high-temperature properties for silicon nitride ceramics required the use of the minimum amount of sintering additives, using pure powders to reduce impurity segregation at the GB or by the formation of a composite microstructure to cause GB pinning.

3 CHEMICAL COMPATIBILITY OF SI-SIALON

A. Hendry (University of Strathclyde, UK) discussed the chemical compatibility of SiC-sialon composites. SiC and Si_3N_4 are both relatively stable and do not react together at high temperatures so that it had been assumed that SiC with sialon do not react together; this is not so and a reaction does occur. The example given by Hendry was:



The reaction becomes important where a sialon acts as a binder for SiC refractories; typically 80-percent SiC and 20-percent sialon. The degree of reaction between sialon and SiC can be followed by the evolution of CO. The reaction can be reduced by sintering in CO above the equilibrium pressure; e.g., 20-30 percent CO at $\sim 1700^\circ C$. It has not yet been possible to obtain complete densification, and experiments are continuing.

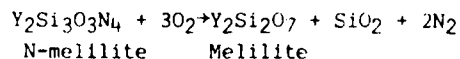
4 GAS PRESSURE SINTERING

C. Ziegler (Deutsche Forschungs und Versuchsanstalt für Luft und Raumfahrt [DFVLR], Cologne, West Germany) discussed their 2-year studies of gas pressure sintering of silicon nitride. The gas pressure suppresses the decomposition of Si_3N_4 so that higher sintering temperatures are possible, less additives are required, and the Si_3N_4 should be usable up to its intrinsic temperature limit of $\sim 1800^\circ C$. A two-step gas sintering process is undertaken. In the first temperature step, the pores close, and the second step is essentially hot isostatic pres-

sure (HIP) sintering. There are many parameters to vary: the amount of Y_2O_3 and Al_2O_3 additives, the various processing steps, and powder characteristics. Ziegler described his typical one-step process, when gas pressure sintering is carried out at $\sim 1875^\circ C$ at 500 bar. A typical two-step sintering involves first, $1875^\circ C$, 90 minutes, 5 bar N_2 , followed by $1925^\circ C$, 60 minutes, 100 bar, when ~ 100 -percent dense Si_3N_4 has been obtained. The material used is LC12-grade (H.C. Stark-Berlin) Si_3N_4 , with $5Y_2O_3 + 1.1 Al_2O_3$. The two-step process reduced the number of residual pores at higher sintering temperatures with no decomposition, and with some increase in grain size, used lesser amounts of additives, particularly Al_2O_3 , to obtain a high-density product.

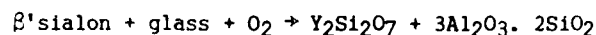
5 OXIDATION PROBLEMS OF NITROGEN CERAMICS

D.P. Thompson, (University of Newcastle-upon-Tyne, UK) discussed the oxidation problem of yttria-densified nitrogen ceramics. These materials have very good properties apart from the fact that they undergo catastrophic oxidation at $\sim 1000^\circ C$, when the material falls apart. This has been considered to be due to the formation of melilite. Thus,



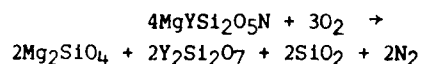
In this reaction, there is a volume increase ΔV of 33 percent, mainly due to the formation of the low-density SiO_2 . In N-ceramics with ~ 15 -percent additions of Y_2O_3 , the overall volume change $\Delta V/V$ is ~ 5 percent. The melilite formed above $1000^\circ C$ is located at the GB, and causes cracking. If there is glassy material at the GB, the cracking is less pronounced because the glass can flow elastically at the melilite formation temperature. Thompson set out to explain how melilite itself is not the culprit for the catastrophic oxidation failure; rather, he reported that the large volume change causes the trouble. Thompson attempted to estimate the maximum $\Delta V/V$ that the material can tolerate without

catastrophic cracking. He considered various reactions; with $\Delta V/V$ 6 percent, cracking results, and similarly at 4 percent. However, when $\Delta V/V < 3$ percent, less cracking occurs, and this may be an acceptable value. This is the case for the reaction:



This reaction has an overall volume change $\Delta V/V$ of 3 percent. This can be understood since the glass is of low density while the mullite is a higher density structure where the Al atoms are 6-coordinated. By avoiding the formation of low-density SiO_2 , the net overall volume change need not be too great.

Another reaction Thompson discussed was:



Here, ΔV is 15 percent, but $\Delta V/V$ is 2.1 percent. In this case, the cracking is very reduced.

When sintering Si_3N_4 in air with 10-percent Y_2O_3 and 2-percent La_2O_3 , Thompson reported no cracking. However, additions of 10-percent Y_2O_3 + 14 percent Nd_2O_3 caused catastrophic cracking. A small addition of alumina removes the problem by forming a sialon glass which can accept the volume change. Again, the alumina may cause mullite rather than SiO_2 to be the product, and since the mullite has a higher density than SiO_2 , the overall volume change is kept small.

Thompson concluded that oxidation cracking in general is not necessarily due to the presence of melilite but rather to the overall volume change on oxidation. If this is kept below 3 percent, catastrophic oxidation cracking can be avoided. The presence of glassy phases at the GB also help by accepting the strains elastically.

6 TRANSPARENT NITROGEN GLASSES

P. Korgul (University of Newcastle-upon-Tyne, UK) has shown that nitrogen glasses are, in fact, transparent. So

far, N-glasses, where up to 50 percent N can replace O, have always been found to be opaque. The N-glasses have a higher hardness, better mechanical properties, lower thermal expansion coefficients, improved leaching characteristics, and an electrical conductivity increased by two orders of magnitude. The N-glass region of the Si-Mg-Al-O-N equilibrium diagram at 1700°C has been determined. The opaque nature is due to precipitates (ppt) being formed from Fe impurities and these ppt act as light-scattering centers. If Fe impurities are excluded, there are no ppt, no scattering centers, and the glass is found to be inherently transparent. The ppt formation has been examined by electron microscopy, and these studies are continuing.

7 OTHER TOPICS

An interesting talk was given by J.J. Pomery (National Institute of Higher Education, Limerick, Ireland) on the pressureless sintering of silicon nitride with mixed oxide additives such as $\text{MgO}+\text{Nd}_2\text{O}_3$ or $\text{MgO}+\text{Y}_2\text{O}_3$. H. Schubert, (Max Plank Institute, Stuttgart, West Germany) discussed the preparation of multiphase ceramic bodies. He considered $\text{ZrO}_2+\text{Y}_2\text{O}_3$ where it is necessary to have powders of small particles sizes, with a uniform distribution of Y_2O_3 . Methods of obtaining uniform distributions were reviewed: co-ppt, spray solutions, use of alkoxides, and hydrothermal ppt of hydroxides followed by freeze-drying.

8 SUMMARY

This meeting gave an opportunity for much discussion between UK and German ceramists. There has been some consideration about holding joint meetings between all of the European ceramic societies, and there were some special discussions to this effect. It may evolve that bi-annual meetings of the European societies will be held, possibly under the auspices of the Science of Ceramic meetings, which

are held each year in a different European country. The next "Science of Ceramics" meeting is planned for 7 through 9 September 1987 at the University of Canterbury in Kent; it is being organized by the Institute of Ceramics, UK.

9 REFERENCE

Lorriman, J., "ICHIBAN-The Japanese Approach to Engineering Education", *Electronics and Power*, (August 1986) 573-577

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